

STANDARD OPERATING PROCEDURE

No. 2420.6E

SAMPLE CONTAINER SELECTION, PRESERVATION AND HOLDING TIMES

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Attachments

1. Guide for Sample Container Selection, Sample Preservation, and Holding Times
Total number of pages: 5.
 2. Guide for Selecting Intermediate Sample Container Material
Total number of pages: 3.
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A. PURPOSE AND APPLICABILITY

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for the selection of the proper sample containers and intermediate sample collection containers or devices when collecting samples for specific constituents (parameters) or groups of constituents; and for determining sample preservation and the holding times of samples from the time of collection until analysis is performed.

The guidance contained herein is applicable to all personnel who collect environmental samples for analysis by the Environmental Services Division (ENSV), including EPA and contractor personnel.

B. DEFINITIONS

| | |
|-------|---|
| ASR | Analytical Services Request |
| BNA | Base-Neutral/Acid Extractable |
| BOD | Biochemical Oxygen Demand |
| CBOD | Carbonaceous Biochemical Oxygen Demand |
| CFR | Code of Federal Regulations |
| DBCP | 1,2-Dibromo-3-chloropropane |
| DO | Dissolved Oxygen |
| EDB | Ethylene Dibromide |
| ENSV | Environmental Services Division |
| GFF | Glass Fiber Filter |
| HEM | Hexane Extractable Material |
| MS | Matrix Spike |
| MSD | Matrix Spike Duplicate |
| NPDES | National Pollutant Discharge Elimination System |
| PCB | Polychlorinated Biphenyl |
| PE | Performance Evaluation |
| PM | Project Manager |
| QC | Quality Control |
| RLAB | Regional Laboratory Branch |
| RSTC | Regional Science and Technology Center (facility where the RLAB is located) |
| RSCC | Regional Sample Control Coordinator |
| SOP | Standard Operating Procedure |
| SSR | Sampling Supplies and QC/PE Samples Request |
| SW | Solid Waste |
| TCLP | Toxicity Characteristic Leachate Procedure |
| TKN | Total Kjeldahl Nitrogen |

| | |
|-----|------------------------------|
| TOX | Total Organic Halogens |
| TPH | Total Petroleum Hydrocarbons |

C. **SAMPLE CONTAINERS**

1. The use of the proper sample container is extremely important to ensure the representativeness of the analytical data obtained, the sufficiency of the sample volume for analysis, and the non-interference or contamination of the sample resulting from the sample container material. When considering the sufficiency of the sample volume for analysis, any matrix spike and matrix spike duplicates (MS/MSD) must be accounted for, and the sampling volume should be adjusted as necessary (One sample for each analyte in each sampling event should at least be double the normal volume or larger volumes for all samples could be used. For example, since water samples for extractables are being collected in 1-gallon bottles, there should be sufficient volume to perform MS/MSD analysis on any sample in the batch.)
2. Special care will be taken to avoid any inadvertent contamination of sample containers prior to or during the sample collection process. Specifically:
 - a. Sample containers shall be left sealed or containerized during storage and transport to the sampling location and until the time of actual sample collection. [Exception: Polyethylene cubitainers are received from the manufacturer with the screw caps not attached. The cubitainers are collapsed and nested. The caps are screwed on to the cubitainers after they are filled with sample.]
 - b. Sample containers will not be rinsed with the media being sampled during the sample collection process unless specifically required for a given parameter or sampling process (e.g., collecting water samples for toxicity testing).

Note: The specific SOPs on sample collection should be referred to for the appropriate collection procedure to be used.

3. The sample container selection process is governed by:
 - a. The parameter or group of parameters to be analyzed; this includes the desired level of detection in many cases.
 - b. The media or matrix to be sampled (i.e., air, solid, tissue, water or other).

- c. The analytical method to be used for the analysis.
 - d. The laboratory (i.e., EPA Region 7 laboratory or a contract laboratory) to perform the analysis. The laboratory where samples will be analyzed is determined by the Laboratory Branch (RLAB) upon receipt of an Analytical Services Request (ASR) form.
- 4. Attachment 1 provides specific guidance for use in Region 7 in selecting the proper sample container by parameter, or parameter group, and the media being sampled. Additional guidance or requirements for acceptable materials of sample containers are contained, by the parameter to be analyzed, in the current 40 CFR Parts 136 and 141, and SW-846. Also, guidelines are normally found in the specific analytical methods and sampling procedures.
 - 5. When the use of intermediate sample collection containers is necessary, guidance on recommended intermediate container materials may be found in Attachment 2.

D. SAMPLE PRESERVATION

- 1. The immediate on-site analysis of samples at the time of collection is, in most cases, neither possible nor practical. Therefore, methods have been established to maintain the integrity of the sample until analysis can be accomplished. Even when samples are preserved in an appropriate manner, they should be analyzed as soon as possible after collection. An integral part of preservation is the selection of the proper sample container, the pretreatment of a sample container (if necessary), and the holding time allowable prior to analysis.
- 2. The purpose of sample preservation is to 1) retard biological action; 2) retard hydrolysis of chemical compounds and complexes; 3) reduce volatility of constituents; and 4) reduce absorption effects. The preservation methods used are generally limited to pH control, chemical addition, refrigeration or freezing. As a rule, the refrigeration (or icing) of samples should be utilized to maintain the samples at a temperature of $4 \pm 2^{\circ}\text{C}$ during sample collection (including the collection of time or flow-weighted composite samples), transport, and storage.
- 3. The current guidance for sample preservation for use in Region 7 is provided in Attachment 1. Although taken into consideration when preparing this guidance, additional specific guidelines and requirements for sample preservation may be found in regulations (e.g., 40 CFR Part 136), publications (e.g., SW-846) and applicable analytical methods.

4. The following guidance is provided for field personnel to use when preserving the types of samples indicated:
 - a. Grab Samples: The applicable preservation method must be accomplished immediately upon sample collection.
 - b. Manually Composited Water Samples: The applicable preservation must be added, in full, to the initial aliquot and thus be available for each subsequent aliquot.
 - c. Automatically Composited Water Samples:
 - (1) When collected for either a single parameter or a parameter group where the type and amount of preservative required are identical, the applicable preservative is added to each container receiving an aliquot, prior to compositing.
 - (2) When collecting a composite sample that will later be split to create samples for a variety of individual parameters and each of these parameters requires different preservation methods, the samples collected for the composite should be iced to maintain a temperature of 4°C until the compositing and splitting can be completed. The appropriate preservative is then added at the time the composite is split into separate containers.
5. Samples of the following media will not be preserved with the addition of any chemical compound, but will be chilled to 4°C after collection and during transport and storage.
 - a. Solids: soil, sediment, sludge
 - b. Tissue (or freeze, -15 to -20°C)
 - c. Other: non-aqueous solutions, product samples (liquid or solid), drum samples, wipe samples
6. The following parameters require special procedures:
 - a. **Biochemical Oxygen Demand (BOD)/Carbonaceous Biochemical Oxygen Demand (CBOD).** Water samples of chlorinated effluents collected for analysis of this parameter must be labeled with the word "CHLORINATED" on the sample tag to alert laboratory personnel.

Chlorinated samples require different analytical procedures than unchlorinated samples for this parameter.

- b. **Cyanide, Total and Amenable to Chlorination.** Water samples for these parameters should not be collected using automatic samplers, but should be collected manually either as a grab or a composite of several grab samples which are preserved at the time of collection. Since oxidizing agents such as chlorine decompose many cyanides, the sample must be treated to eliminate such agents, if they are present, at the time of collection. The presence of chlorine is determined by testing a drop of the sample with potassium iodide (KI)-starch test paper. A change in the color of the paper to blue indicates the need for treating the sample with a dechlorination agent. This treatment is accomplished by adding ascorbic acid, a few crystals at a time followed by the subsequent testing of a drop of sample until no color is produced on the KI indicator paper. An additional 0.6 gram of ascorbic acid is then added for each liter of sample volume. Preservation of the sample is then accomplished by adding 2 mL of 10 N sodium hydroxide solution or 10 pellets of sodium hydroxide crystals per liter of sample (to pH \geq 12) and by icing the sample to 4°C during transport and storage.
- c. **Dissolved Oxygen (DO).** Water samples for this parameter are collected only on a grab basis. When collecting a sample for this parameter, the sample bottle should be filled to overflowing to ensure that no air bubbles are entrapped in the bottle when the stopper is replaced. When immediate measurement is not possible on site utilizing the DO probe method, the sample will be "fixed" immediately upon collection by first adding 2 ml of manganous sulfate (MnSO_4) solution and then 2 ml of alkali-iodide-azide solution well below the surface of the liquid. The sample is mixed by inverting the bottle several times while holding the stopper in place and allowing to set until the floc has settled half way. Carefully remove the stopper and immediately add 2 ml of concentrated sulfuric acid (H_2SO_4) by allowing the acid to run down the neck of the bottle. Re-stopper and mix again and store at 10-20°C out of direct sunlight. Completion of the analysis for DO utilizing the Winkler titration method (Azide Modification) should be accomplished as soon as possible after collection and fixing, but not more than 8 hours after collection.
- d. **Metals, Dissolved.** Water samples for this parameter must be filtered on site utilizing a 0.45 μm membrane filter as soon as practical after collection and then acidified with 5 mL 1:1 HNO_3 per liter of sample.

- e. **Microbiology (Total and Fecal Coliform; Fecal Streptococci).** Water samples for these parameters will be collected only on a grab basis. For chlorinated effluents, sodium thiosulfate (0.008% $\text{Na}_2\text{S}_2\text{O}_3$) is added to the sample. This dechlorinating agent is normally added during sample container preparation and is, therefore, normally present in the sample container. Care must be taken during sample collection to avoid overfilling or rinsing out the agent. In addition, care must be taken to avoid contamination of the sterile sample container prior to or during sample collection; i.e., leave cap on container until ready to collect sample and do not place fingers in container or on the inside of the cap, while collecting a sample. An air space should be left at the top of the container after the sample is collected. Ice to 4°C.
- f. **Oil and Grease (Hexane Extractable Material, HEM)** Water samples for this parameter will be collected in one liter glass bottles on a grab basis only and acidified with 1:1 HCl to pH < 2 immediately after collection. The sample container should never be rinsed with the water or wastewater because these constituents tend to adhere to the sides of the container. Care should be taken to avoid contamination of the sample from fingers placed in the container or on the inside liner of the cap. In addition, enough air space should be allowed in the container to allow for the addition of the preservative.
- g. **Organics, Volatiles.** Grab samples only are collected for these parameters. Each sample will consist of two (2) 40-mL vials. Generally, four (4) 40-mL vials per water sample will be collected for low detection level and drinking water samples.
- (1) Drinking water samples containing residual chlorine must be treated with sodium thiosulfate or ascorbic acid (depending on the analytical method) at the time of collection.
 - (2) Wastewater samples containing residual chlorine must be treated with ascorbic acid (25 mg per 40 mL) at the time of collection. These dechlorinating agents must be placed in the vials prior to collecting the samples.
 - (3) When collecting water samples, fill the sample bottles to overflowing, but take care not to flush out the sodium thiosulfate or ascorbic acid, if present. No air bubbles should pass through the sample as the bottle is filled, or be trapped in the sample when the bottle is sealed. After collection, the pH of the sample is adjusted

to a pH < 2 by carefully adding one drop of 1:1 HCl for each 20 mL of sample volume. Seal the sample bottles teflon-face down, and shake vigorously for 1 minute. A proper seal can be checked by inverting the sample and lightly tapping the end on a solid surface. If air bubbles are present, open the vial, add additional sample, reseal, and recheck for air bubbles. Store samples out of direct sunlight.

- h. **Phenols (Phenolics).** Water samples for this parameter should not be collected by using automatic samplers, but should be collected as a grab or a manual composite of grabs and preserved with 2 mL H₂SO₄ to a pH < 2 at the time of collection. The samples should be iced to maintain them at 4°C during transport and storage.

E. SAMPLE HOLDING TIMES

1. The issue of holding times for samples is critical in the sample collection and analysis process, because the integrity of the samples can be affected depending on the parameter to be analyzed. Sample holding times are defined as the period of time between sample collection and initiation of sample analysis. In the case of timed composite samples, the holding time starts at the end of the compositing period (i.e., at the time the last portion of the composite sample is obtained).
2. Since holding times can affect the validity of the reported analytical results (especially in certain media programs and in enforcement actions), everyone involved in planning and executing sampling activities; planning and performing analyses; and reviewing analytical results must be cognizant of the implications of exceeding them during the process. In many instances, the holding times are required by specific regulations (e.g., 40 CFR Part 136 for wastewater samples under the NPDES program), while many others are recommended. Also, see Footnote 8 of Attachment 1.
3. Although many of the holding times contained in Attachment 1 were derived from regulatory requirements, the holding times should be considered as guidelines. When making decisions on the validity of analytical results based on holding times, personnel should consult appropriate regulations to determine if there are specific requirements for sample holding times.

F. OBTAINING SAMPLING SUPPLIES

1. When sampling supplies (e.g., sample containers, sample collection devices, preservatives, etc.) are needed, the project manager for the specific sampling

activity requests the necessary supplies on the Sampling Supplies and QC/PE Samples Request (SSR).

2. The requestor can pick up the supplies at the ENSV warehouse facility (3150 Dodge). The preservatives must be picked up at the RSTC (300 Minnesota Avenue). It is recommended that the requestor contact the Regional Sample Control Coordinator (RSCC) or designated back-up at the RLAB before going to either facility to ensure the supplies are ready for issuance.

G. **REFERENCES**

1. Code of Federal Regulations, Title 40 (40 CFR), Part 136.
 2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), U.S. EPA.
 3. Standard Methods for the Examination of Water and Wastewater, Joint Editorial Board, American Public Health Association, American Water Works Association and Water Environment Federation, Latest Edition.
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Attachment 1**Guide for Sample Container Selection, Preservation and Holding Times**

| Parameter ¹ | Sample Container ² | Preservation | Holding Time ³ |
|---|---|-------------------------|---------------------------|
| I. Air and Gaseous | | | |
| Dioxins/Furans | Puff glass jar | Cool 4°C | 365 days |
| Metals on High-Vol GFF | 1-gal. resealable plastic bag | Ambient Temperature | |
| Ozone Precursors | | | |
| Ambient levels | 6-L canister | None | 60 days |
| Source levels | 400 mL canister | None | 60 days |
| Particulate Matter on High-Vol GFF | 1-gal. resealable plastic bag | Ambient Temperature | |
| Pesticides/PCBs | Puff glass jar | Cool 4°C | 7 days |
| Semivolatiles/BNA | Puff/XAD glass jar | Cool 4°C | 14 days |
| Volatile Organics | | | |
| Ambient levels | 6-L canister | None | 60 days |
| Source levels | 400 mL canister | None | 60 days |
| GC/MS Scan | 6-L canister | None | 30 days |
| II. Soil, Sediment and Solids | | | |
| A. Non-Product Samples | | | |
| Asbestos | Plastic jar | None | None |
| Cyanide | 8-oz. glass jar | Cool 4°C | 28 days |
| Dioxin/Furans | 8-oz. glass jar/sealable plastic bag in 1-qt. paint can | Cool 4°C | 365 days |
| Explosives | 8-oz. glass jar | Cool 4°C | 14 days |
| Flashpoint | 8-oz. glass jar | Cool 4°C | |
| GC/MS Scan, SemiVOA | 8-oz. glass jar | Cool 4°C, store in dark | 14 days |
| GC/MS Scan, VOA | 2, 40mL VOA vials | Cool 4°C | 14 days |
| Herbicides | 8-oz. glass jar | Cool 4°C | 14 days |
| Metals | | | |
| All metals except Cr ⁺⁶ and Hg, collected separately | 8-oz. glass jar | Cool 4°C | 180 days |
| Mercury | 8-oz. glass jar | Cool 4°C | 180 days |
| Cr ⁺⁶ | 8-oz. glass jar | Cool 4°C | 30 days |
| Methanol | 8-oz. glass jar | Cool 4°C | 14 days |
| Nutrients | | | |
| Nitrogen (NH ₃ , NO ₂ , TKN) | 8-oz. glass jar | Cool 4°C | 28 days |
| Phosphorous, total | 8-oz. glass jar | Cool 4°C | |
| Oil and Grease | 8-oz. glass jar | Cool 4°C | |
| pH | 8-oz. glass jar | Cool 4°C | None |
| Phenolics (colorimetric) | 8-oz. glass jar | Cool 4°C | 28 days |
| Perchlorate | 8-oz. glass jar | Cool 4°C | 28 days |
| Pesticides/PCBs | 8-oz. glass jar | Cool 4°C | 14 days |
| Radioactivity | 8-oz. glass jar | Cool 4°C | 180 days |

| Parameter | Sample Container | Preservative | Holding Time |
|--|--|--|---|
| II. Soil, Sediment, and Solids (cont.) | | | |
| A. Non-Product Samples (cont.) | | | |
| Semivolatiles/BNA | 8-oz. glass jar | Cool 4°C | 14 days |
| Sulfate/Sulfide | 8-oz. glass jar | Cool 4°C | 28 days |
| Soil Toxicity Test | 1-gal. ziplock bag | Cool 4°C | 56 days |
| Total Organic Carbon | 8-oz. glass jar | Cool 4°C | 28 days |
| Total Petroleum Hydrocarbons (TPH) | 8-oz. glass jar | Cool 4°C | 14 days |
| SemiVOA | | Cool 4°C | 14 days |
| VOA | 2, 40-mL glass vials | Cool 4°C | 14 days |
| Total Kjeldahl Nitrogen | 8-oz. glass jar | Cool 4°C | None |
| Organic Parameters | 8-oz. glass jar | Cool 4°C | 14 days |
| All except volatile organics | | Cool 4°C or MeOH & Cool 4°C or NaHSO ₄ & Cool 4°C | 14 days |
| Volatile organics ³ | 2, 40-mL glass vials | Cool 4°C or MeOH & Cool 4°C or NaHSO ₄ & Cool 4°C | 14 days |
| TCLP | 8-oz. glass jar | Cool 4°C | 180 days to extract, 180 days after extraction (required) |
| Metals, except Hg | | Cool 4°C | 28 days to extract, 28 days after extraction (required) |
| Mercury | 8-oz. glass jar | Cool 4°C | 14 days to extract, 14 days after extraction (required) |
| Volatile organics | 2, 40-mL glass vials | Cool 4°C | 14 days to extract, 7 days to extraction, 40 days after extraction (required) |
| Semivolatile organics | 8-oz. glass jar | Cool 4°C | 14 days |
| Pesticides/Herbicides | 8-oz. glass jar | Cool 4°C | 14 days |
| B. Product Samples | | | |
| All parameters | 8-oz. glass jar/sealable plastic bag in paint can | Cool 4°C | |
| III. Tissue | | | |
| Fish, collected for whole body/edible portion, all parameters | Double wrapped in heavy duty foil | Freeze | |
| Resectioned tissue, collected for: | Double wrapped in heavy duty foil | Freeze | 180 days |
| Metals | Double wrapped in heavy duty foil | Freeze | |
| Semivolatiles | Double wrapped in heavy duty foil | Freeze | |
| Volatiles | Double wrapped in heavy duty foil | Freeze | |
| Foliage | Double wrapped in heavy duty aluminum foil | Freeze | |
| Herbicides/Pesticides | | Freeze | |
| Macroinvertebrates ⁶ | | 70% ethanol | 6 months |
| Periphyton ⁶ | | Freeze, store in dark | 30 days |
| Chlorophyll A | | 5% formalin, Cool 4°C, store in dark | 6 months |
| Enumeration | | | |
| Dioxins/Furans | Double wrapped in heavy duty foil | Freeze | 365 days |
| Phytoplankton, collected for ⁶ | | Cool 4°C, store in dark | 14 days |
| Chlorophyll A | | 5% formalin | 6 months |
| Enumeration | | | |

| Parameter | Sample Container | Preservative | Holding Time |
|---|---|---|--|
| IV. Aqueous Samples | | | |
| Chlorine Dioxide | 1-L plastic cubitainer | Cool 4°C | None |
| Chlorophyll A | 4-L plastic cubitainer | Cool 4°C, store in dark | 14 days |
| Coliform, fecal | 300-mL sterile plastic bottle | Cool 4°C, 0.008% Na ₂ S ₂ O ₃ | 6 hours |
| Dioxins/Furans | 1-L amber glass bottle | Cool 4°C | 365 days |
| Dissolved Organic Carbon | 1-L amber glass bottle | H ₂ SO ₄ to pH<2 Cool 4°C | 28 days |
| Explosives | 128-oz. amber glass bottle | Cool 4°C | 7 days to extract, 40 days after extraction |
| Herbicides | 128-oz. amber glass bottle | Cool 4°C | 7 days to extract, 40 days after extraction |
| Flashpoint | 8-oz. glass jar | Cool 4°C | |
| Metals (except Hg and Cr ⁺⁶) | | | |
| Total and acid soluble | 1-L plastic cubitainer | HNO ₃ to pH<2 | 6 months |
| Dissolved | 1-L plastic cubitainer | Filter HNO ₃ to pH<2 | 6 months |
| Chromium, hexavalent | 1-L plastic cubitainer | Cool 4°C | 24 hours |
| Mercury | 1-L plastic cubitainer | HNO ₃ to pH<2 | 28 days |
| Strontium | 1-L plastic cubitainer | HNO ₃ to pH<2 | 6 months |
| Acid, % | 1-L plastic cubitainer | Cool 4°C | None |
| Alkalinity | 1-L plastic cubitainer | Cool 4°C | 14 days |
| BOD/CBOD | 1-L plastic cubitainer | Cool 4°C | 48 hours |
| COD | 1-L plastic cubitainer | H ₂ SO ₄ to pH<2 Cool 4°C | 28 days |
| Chlorine (residual) | 1-L plastic cubitainer | Cool 4°C | 1 day |
| Conductivity | 1-L plastic cubitainer | Cool 4°C | 28 days |
| Cyanide (total and amenable To chlorine) | 1-L plastic cubitainer | (Ascorbic acid), NaOH to pH>12, Cool 4°C | 14 days |
| Halides (Br, Cl, F) | 1-L plastic cubitainer | Cool 4°C | 28 days |
| Haloacetic Acids/Dalapon | 1-L amber glass bottle | Ammonium Chloride, Cool 4°C | 14 days |
| Hardness | 1-L plastic cubitainer | HNO ₃ to pH<2, Cool 4°C | 6 months |
| Inorganic Anions | 1-L plastic cubitainer | EDA, Cool 4°C | 14 days |
| Methane, Ethane, Ethene | 2, 40-mL VOA vials | Cool 4°C | 7 days |
| Methanol | 1-L amber glass bottle | Cool 4°C | 7 days |
| Nonfilterable Solids (NFS) | 1-L plastic cubitainer | Cool 4°C | 7 days |
| Oxygen, Dissolved (Winkler) | 300-mL glass BOD bottle with Attached ground glass stopper | MnSO ₄ + Alkali- Iodide-Azide, H ₂ SO ₄ | 8 hours |
| Dissolved Oxygen, probe Method | 1-L plastic cubitainer | Cool 4°C | 1 day |

| Parameter | Sample Container | Preservative | Holding Time |
|------------------------------------|----------------------------|---|--|
| IV. Aqueous Samples (cont.) | | | |
| pH | 1-L plastic cubitainer | Cool 4°C | determine immediately |
| Perchlorate | 1-L plastic cubitainer | Cool 4°C | 28 days |
| Residue | | | |
| All but settleable | 1-L plastic cubitainer | Cool 4°C | 7 days |
| Settleable | 1-L plastic cubitainer | Cool 4°C | 48 hours |
| Sulfate | 1-L plastic cubitainer | Cool 4°C | 28 days |
| Sulfide | 1-L plastic cubitainer | Zinc acetate + NaOH to pH>9, Cool 4°C | 7 days |
| Total Dissolved Solids (TDS) | 1-L plastic cubitainer | Cool 4°C | 7 days |
| Total Kjeldahl Nitrogen | 1-L plastic cubitainer | H ₂ SO ₄ to pH<2.5, Cool 4°C | 28 days |
| Total Solids | 1-L plastic cubitainer | Cool 4°C | 7 days |
| Turbidity | 1-L plastic cubitainer | Cool 4°C | 48 hours |
| Nutrients | | | |
| Nitrogen-Ammonia | 1-L plastic cubitainer | H ₂ SO ₄ to pH<2, Cool 4°C | 28 days |
| Nitrogen-Organic | 1-L plastic cubitainer | H ₂ SO ₄ to pH<2, Cool 4°C | 28 days |
| Nitrate | 1-L plastic cubitainer | Cool 4°C | 48 hours |
| Nitrate-Nitrite | 1-L plastic cubitainer | H ₂ SO ₄ to pH<2, Cool 4°C | 28 days |
| Nitrite | 1-L plastic cubitainer | Cool 4°C | 48 hours |
| Phosphorous (total) | 1-L plastic cubitainer | H ₂ SO ₄ to pH<2.5, Cool 4°C | 28 days |
| Ortho-phosphate | 4-oz. plastic bottle | Filter, Cool 4°C | 48 hours |
| Phosphorous, (dissolved) | 1-L plastic cubitainer | Filter, H ₂ SO ₄ to pH<2, Cool 4°C | 28 days |
| Carbamates ¹⁹ | 60-mL screw cap vial | 1.8 mL monochloroacetic acid buffer pH<3, Cool 4°C | 14 days |
| Oil and Grease (HEM) | 1-L glass jar | 1:1 HCL to pH<2, Cool 4°C | 28 days |
| Pesticides/PCBs | 128-oz. amber glass bottle | Cool 4°C | 7 days to extract, 40 days after extraction ⁹ |
| Phenolics | 1-L glass jar | H ₂ SO ₄ to pH<2, Cool 4°C | 28 days |
| Radionuclides | 1-L plastic cubitainer | HNO ₃ to pH<2 | 6 months (water:alpha/beta) 5 days(gamma; DW:alpha/beta) |
| Semivolatile/BNA | 128-oz. amber glass bottle | Cool 4°C, store in dark | 7 days to extract, 40 days after extraction |
| GC/MS Scan (BNA) | 128-oz. amber glass bottle | Cool 4°C, store in dark | 7 days |
| Total Organic Carbon (TOC) | 1-L amber glass bottle | H ₂ SO ₄ to pH<2, Cool 4°C | 28 days |
| Total Organic Halogens (TOX) | 8-oz. amber glass bottle | Cool 4°C | 7 days |

| Parameter | Sample Container | Preservative | Holding Time |
|-------------------------------------|---|--------------------------------|---|
| IV. Aqueous Samples (cont.) | | | |
| Total Petroleum Hydrocarbons (TPH) | | | |
| SemiVOA | 128-oz. amber glass bottle | Cool 4°C | 7 days |
| VOA | 2, 40-mL VOA vials | Cool 4°C | 14 days |
| Toxicity Tests | | | |
| Acute | 10-L (2.5-gal) plastic cubitainer | Cool 4°C | 36 hours |
| Bioscreen (24 or 48 hr.) | 4-L (1-gal.) plastic cubitainer or glass bottle | Cool 4°C | 24 hours |
| Chronic | 20-L (2.5/5-gal.) plastic cubitainer | Cool 4°C | 36 hours |
| Triazine Herbicides | 128-oz. amber glass bottle | Cool 4°C | 14 days |
| Tritium | 8-oz. glass jar | None | 5 days |
| Volatile Organics ^{1,5} | | | |
| Purgeable halocarbons | 2, 40-mL glass vials | HCl to pH<2, Cool 4°C | 14 days |
| Purgeable aromatic hydrocarbons | 128-oz. amber glass bottle | HCl to pH<2, Cool 4°C | 7 days |
| Routine Detection Level | 2, 40-mL glass vials | HCl to pH<2, Cool 4°C | 14 days |
| Low Detection Level | 4, 40-mL glass vials | HCl to pH<2, Cool 4°C | 14 days |
| EDB/DBCP | 2, 40-mL glass vials | Sodium Thiosulfate Cool 4°C | 14 days |
| GC/MS Scan (VOA) | 2, 40-mL VOA vials | HCl to pH<2, Cool 4°C | 14 days |
| V. Liquid, Non-Aqueous | | | |
| TCLP (> 5% solids) ⁴ | | | |
| Mercury | 8-oz. glass jar | Cool 4°C | 28 days to TCLP extract, 28 days after extraction |
| Metals, except Hg | 8-oz. glass bottle | Cool 4°C | 180 days |
| Volatile Organics | 2, 40-mL glass vials | Cool 4°C | 14 days to TCLP extract, 14 days after extraction |
| Semivolatile Organics | 8-oz. glass jar | Cool 4°C | 14 days to TCLP extract, 7 days to extraction, 40 days after extraction |
| Pesticides/Herbicides | 128-oz. amber glass bottle | Cool 4°C | 7 days |
| Organic Parameters | | | |
| All except volatile organics | 8-oz glass jar | Cool 4°C | 7 days to extract, 40 days after extraction |
| Volatile organics | 8-oz glass jar | Cool 4°C | 14 days |
| VI. Wine Samples⁷ | | | |
| Arsenic | 8-oz. glass jar | Cool 4°C | |
| Cyanide | 8-oz. glass jar | Cool 4°C | |
| Dioxin | 8-oz. glass jar | Cool 4°C | 365 days |
| Herbicides/Pesticides | 8-oz. glass jar | Cool 4°C | 14 days |
| Metals | 8-oz. glass jar | Cool 4°C | 180 days |
| Picric Acid | 8-oz. glass jar | Cool 4°C | |
| Organic other parameters | | | |
| All except volatile organics | 8-oz. glass jar | Cool 4°C | 7 days to extract, 40 days after extraction |
| Volatile organics | 8-oz. glass jar | Cool 4°C | |

| Parameter | Sample Container | Preservative | Holding Time |
|--|------------------|--------------|--|
| VI. Wipe Samples (cont.) | | | |
| TCLP (> 0.5% solids) Metals, except mercury | 8-oz. glass jar | Cool 4°C | 180 days to TCLP extract, 180 days after extraction |

¹ Non-product and product sample definitions: A non-product sample is a sample which consists primarily of naturally occurring materials that may contain mechanically or chemically manufactured materials or substances as contaminants. A product sample is a sample which is known to consist primarily of a mechanically or chemically manufactured material that does not otherwise occur naturally in the immediate environment being sampled. A sample which cannot be identified as a non-product sample should be considered a product sample.

² All glass containers require a Teflon-lined lid or cap.

³ These parameters are always collected as replicates. The sample container consists of two or four 40-mL glass vials (VOA vials) and an activated carbon filled thimble contained in a 1-L plastic cubitainer.

⁴ § 40 CFR Part 261 Appendix II, 2.1: For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μ m glass fiber filter, is defined as the TCLP extract.

⁵ Sodium Thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) or Ascorbic Acid ($\text{C}_6\text{H}_8\text{O}_6$) is utilized to de-chlorinate samples of chlorinated water or wastewater prior to pH adjustment. For drinking water samples, consult the applicable method to determine appropriate dechlorinating agent.

⁶ Sample containers, preservation, and holding times vary. The information provided is Regional guidance. For compliance samples, consult appropriate references for complete preservation requirements.

⁷ Each jar should contain a medical gauze pad.

⁸ Generally, there are no required holding limits for air, soil, product and wipe media. (Exceptions: see Chapter 4 of SW-846). However, it is recommended that samples be analyzed within the holding time limits established in aqueous media for the specific analytes or analyte groups.

⁹ Method 505 holding time 14 days (7 days for Heptachlor), preserve with Sodium Thiosulfate, 4°C, container 40-mL vial.

¹⁰ Method 531.1 samples must be preserved to a pH<3 using monochloroacetic acid to minimize degradation of oxamyl, 3-hydroxycarbofuran, aldicarb sulfoxide, and carbaryl in neutral and basic waters. If residual chlorine is present add 80 mg of sodium thiosulfate per liter of sample to the sample bottle prior to collecting the sample.

Attachment 2**Guide for Selecting Intermediate Sample Container Material**

| Media Sampled/Parameter | Intermediate Sample Container Material |
|--|--|
| <u>I. Soil, Sediment and Solids</u> | |
| A. Non-Product and Product Samples | |
| All parameters except volatile organics | Glass, Aluminum |
| Volatile Organics ¹ | Glass, Stainless Steel, Aluminum |
| <u>II. Tissue</u> | |
| A. Fish | |
| All parameters | Glass, Plastic |
| <u>III. Liquids</u> | |
| A. Aqueous Samples | |
| Chlorophyll A | Artificial substrate (glass slide) |
| Coliform, fecal ² | Glass or Plastic |
| Dioxin/Furans | Glass, Stainless Steel (solvent rinsed) |
| Explosives | Glass |
| Metals: | |
| All except Cr ⁶⁺ | Glass, Plastic, Automatic sampler equipped with Tygon intake tubing and glass or plastic compositing container. |
| Chromium, hexavalent | Glass, Plastic |
| Minerals and Dissolved Materials: | |
| Acid (%), Alkalinity, BOD, Chloride, Conductivity, Hardness, Residue, Sulfate, Turbidity | Glass, Plastic, Stainless steel, Automatic sampler equipped with Tygon (or Teflon) intake tubing, and glass or plastic compositing container |
| Chlorine | Glass, Plastic, Stainless steel |
| COD | Glass, Plastic, Stainless steel, Automatic sampler equipped with Tygon (or Teflon) intake tubing, and glass or plastic compositing container |
| Cyanide | Glass, Plastic, Stainless steel |

| Media Sampled/Parameter | Intermediate Sample Container Material |
|--|---|
| III. Liquids (cont.) | |
| A. Aqueous Samples (cont.) | |
| Fluoride | Plastic, Automatic sampler equipped with Tygon (or Teflon) intake tubing, and plastic compositing container |
| Oxygen, dissolved | Glass, Plastic, Stainless steel |
| pH, lab or field | Glass, Plastic, Stainless steel, Teflon |
| Sulfide | Glass, Plastic, Stainless steel |
| Nutrients (N & P) | Glass, Plastic, Stainless steel, Automatic sampler equipped with Tygon (or Teflon) intake tubing, and glass or plastic compositing container |
| Oil and Grease ¹ | Glass |
| Pesticides/PCBs | Glass, Stainless steel, Automatic sampler equipped with Teflon intake tubing and glass compositing container (cleaned and solvent rinsed) |
| Phenols/Phenolics | Glass, Plastic, Stainless steel |
| Radionuclides | Glass, Plastic |
| Semivolatiles/BNA | Glass, Stainless steel, Automatic sampler equipped with Teflon intake tubing and glass compositing container (cleaned and solvent rinsed) |
| Total Organic Carbon | Automatic sampler equipped with Tygon (or Teflon) intake tubing, and glass or plastic compositing container |
| Total Organic Halogens | Glass, Plastic |
| Toxicity Tests: Acute Bioscreen Chronic | Automatic sampler equipped with Teflon or new Tygon tubing, and glass or Nalgene compositing container (see SOP No. 2334.6 for tubing and container cleaning) |
| Volatile Organics ¹ | Glass, Stainless steel (not solvent rinsed) |
| B. Non-Aqueous Product Samples | |
| All parameters | Glass |

¹An intermediate sample collection device is not recommended for this parameter, but, if one is necessary, care must be taken to insure that the device has not been solvent rinsed.

²An intermediate container is not recommended for this parameter; therefore, every effort must be made to collect the sample directly into the sample container.